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IEA/ANNEX II POWDER CHARACTERIZATION COOPERATIVE PROGRAM

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ABSTRACT

Materials characterization will play a key role in such critical issues as quality, reliability, and manufacturing cost reduction of emerging high performance ceramic materials. The identification of better analytical standards and standard procedures for obtaining the **true** values of powder characteristics is an important component of this overall thrust. The U.S. Department of Energy, with the Oak Ridge National Laboratory, has initiated an international program to comprehensively characterize Si_3N_4, Si, SiC, and ZrO_2 powders in laboratories in the United States, the Federal Republic of Germany, and Sweden to expedite the accomplishment of these objectives.

INTRODUCTION

The main objective of this program was the interlaboratory comparison of measurements which most closely approach the **true** value of materials characteristics which are suitable for enhanced quality, reproducibility, and reliability. The goal of this cooperative effort among several independent groups was the identification of the most useful characterization methods, as well as standard procedures and reference materials.

PROGRAM POWDER

Selection

Two groups of powders, a primary set and a secondary set, were selected for this program. The primary group of powders, in order of priority, are: GTE (Ube) silicon nitride (Si_3N_4); Kema Nord/IV-D silicon (Si); H. C. Starck (<5 μm) silicon carbide (SiC); and Toyosoda yttria stabilized zirconia (ZrO_2). An internal reference material was used by all participants for instrumental calibration and determination of precision in conducting the tests. This material was H. C. Starck (LC-10) silicon nitride (Si_3N_4) from the Federal Republic of Germany.

A secondary group of powders was also selected by the participants and included the following, in priority order: Kema Nord silicon nitride (Si_3N_4); Lonza (UF-10) silicon carbide (SiC); Elkem Metals silicon (Si); and Toyosoda monoclinic zirconia (ZrO_2).

The powders in each group were reviewed each year, and the Executive Committee, acting by unanimity, had the power to change the powders selected.

Procurement

The powders were obtained by Oak Ridge National Laboratory (ORNL) and delivered to the National Institute of Standards and Technology for sampling, distribution, and archiving. See Table 1 for distribution dates.

Table 1. POWDER DISTRIBUTION SCHEDULE

Powder	Original Date	Current Status
Toyosoda/Y_2O_3 Stabilized ZrO_2	October 1986	Completed
Starck/LC-10 Si_3N_4	November 1986	Completed
GTE (Ube) Si_3N_4	April 1987	Mid-October
Kema Nord/IV-D Si	April 1987	Mid-November
Starck (<5 μm) SiC	February 1987	Late Jan. 1986

Division and Homogeneity Tests of Ceramic Starting Powders

The division of a bulk lot of powder into many samples required the use of probability sampling. Recommended practice for probability sampling is described in ASTM Standard E 105-58. For the preparation of powder samples, a detailed protocol was developed to ensure the use of verifiable procedures, the maintenance of clean operating conditions, and

the use of randomization procedures throughout the operation. The general steps in the procedure were as follows:

1. Bulk powder was blended in a cone blender.

2. First division of powder into sublots was performed manually with the powder distrib uted to eight or more plastic-lined metal containers. The powder was distributed in small increments.

3. A random selection of sublots, vacuum drying, and division of sublots into 8 or 16 samples using a spinning riffler.

4. A random selection of samples vacuum drying and second riffling of samples was carried out.

5. The process was repeated using a microriffler, operated in a glove box, which was flushed with dry argon. The powder riffled directly into glass vials which were sealed with a plastic cap and were stored in a metallized, heat-sealable polymer bag.

6. Each vial was flame sealed in a glass tube to maintain long-term stability of ambient conditions, see Figure 1.

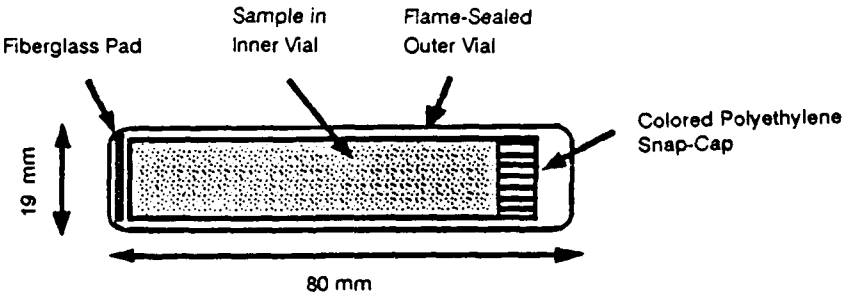

Figure 1. Schematic of flame-sealed sample.

The superiority of the spinning riffler over other sampling techniques was demonstrated by several investigators.[1-6] For sampling of a binary sand mixture, it was found to give a relative standard deviation of an estimated maximum relative sample error of 0.42 percent. Sampling of powders by spinning riffler was recommended in ASTM Standard F 577B-78.

Procedures for preparing samples of approximately 10 g size from bulk lots of powder of 25 kg were tested with commercially available zirconia powder. Samples were measured after

1. ALLEN, T., and KAHN, A. A. *Critical Evaluation of Powder Sampling Procedures.* Chem. Engr. (London), v. 238, CE108-12
2. MONTGOMERY, J. R. *Revolving Riffler for Subdivision Ground Magnesium Chips.* Analyt. Chem. v. 40, no. 8, 1968, p. 1399-1400.
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5. HATTON, T. A. *Representative Sampling of Particles with a Spinning Riffler. Stochastic Model.* Powder Technol., v. 19, 1978, p. 227-233.
6. CHARLIER, R., and GOOSSENS, P. J. D. *Sampling a Heterogeneous Powder Using a Spinning Riffler.* Powder Technol., v. 4, 1970-1971, p. 351-359.

manual division into sublots before final riffling to determine approximate yield and sample-to-sample variability. A comparison of the percent relative standard deviation for these two stages, 1.28% verses 1.70%, indicated that most of the variation in sample size was introduced when the powder was split manually into sublots. The recovery of powder prior to final riffling was 98.7%.

Eight randomly selected vials were submitted for each certification analysis. The following characteristics and methods were used to establish the uniformity of the powder samples:

Bulk density - helium (He) pycnometer
Specific surface area (SAA) - BET
Particle size - photon correlation spectroscopy (PCS) (quasi-elastic light scattering)
Chemical composition:
  Zr, Y, Al - X-ray fluorescence (XRF)
  Total elemental - neutron activation analysis (NAA)
Phase composition - X-ray diffraction (XRD)

Results for the study of the sample variations are summarized in Table 2 for the methods listed above. The results are presented in terms of the relative standard deviation, expressed as a percentage. An estimate of the percent measurement error is also given. The estimated measurement errors are based on replicated measurements for each of the samples. With the exception of the particle size measurements, the coefficients of variation are less than estimated measurement errors which implies that no sample-to-sample variation can be inferred from the measurements.

Table 2. SUMMARY OF CERTIFICATION TESTS

Characteristic	Method	Number of Samples	Relative Standard Deviation (%)	Estimated Measurement Error (%)
Bulk Density	He Pycn.	9	0.3	0.9
Specific SA	3 Pt. BET	9	2	5
Particle Size	PSC	9	5	3
Composition:				
[Y]/[Zr]	XRF	10	0.2	0.3
[Al]/[Zr]	XRF	10	6	10
[monocl]	XRF	10	5	10
Major	NAA	9	0.8	0.7
Minor	NAA	9	2	2
Trace	NAA	9	6	4
Weight of Samples (Stage 3)		96	2	

Major - NAA: neutron activation analysis; major constituents - Zr, Y, Hf
Minor - NAA: minor constituents - Al, Cl, Ca, Ti
Trace - NAA: trace constituents - Na, K, Sc, V, Sm, Eu

The results for the measurement of the particle size distribution suggests that a small amount of sample-to-sample variation was present. This variation was found to be due to a second size mode lying largely outside the range of the PCS instrument which made a, nonetheless, small and uncontrolled contribution to the measurement.

Variation of the chemical composition was not detected within the precision of the instrument.

CHARACTERIZATION METHODOLOGY AND ACQUISITION

Overall Philosophy

In any system involving specific material properties, characteristics of the precursor constituents must first be identified and defined. The extrinsic properties of any material is uniquely defined by its internal chemistry (i.e., phases present), microstructure, and any processing defects.

It is for this reason that analysis of the complete data set of powder characteristics (physical, chemical, and defects) by traditional statistical or logic programming techniques are necessary. Once this data base is established, there can be quantitative correlations made to ceramic characteristics and properties. Ultimately, this information can be utilized for artificial intelligence-type knowledge bases.

Measurements

Characterization methods must be designed to provide sufficient characterization by each participant for interlaboratory data correlation; i.e., particle size and distribution, morphology, major element content, minor element content, trace element content (including volatiles), crystalline and noncrystalline phases, and physical defects such as degree of agglomeration and residual stress. Analysis selection depends on experience and manpower of each working group.

Storage of particle size data is a complex issue. If a log-normal assumption is used (straight line on log-probability paper), then for a single mode powder, only two parameters are needed to describe the powder; if two modes are present, five parameters are needed to describe the powder. Therefore, initially, the program will assume log-normal characteristics for the simple storage of data.

One major problem in quantified physical characterization is the utilization of various measurement techniques for the same parameter. Particle size and particle size distributions are an example of this ambiguity since analysis can be based on several different physical phenomena, each resulting in a different size, and use a variety of representations including weight %, volume %, number %, etc. Therefore, some method of putting the size and distribution data in physically comparable form is very important. There are two possible solutions to this problem: (1) normalization to an internal reference powder, or (2) calculation of previously agreed upon size types using the Hatch-Choate equations.[7]

Internal Reference Powder Normalization

First Stage

a. Each analysis was first performed on the reference powder. The resulting raw data to be collected.

7. HATCH, T., and CHOATE, S. P. *J. Franklin Institute*. 1929, p. 207, 369.

b. Statistical identification of parameter values.

(1) geometric median and
(2) geometric standard deviation.

c. Agreement on standardized values for physical characterization.

Second Stage

a. Begin measurements of other primary powders. Collect all raw data.

b. Assemble raw data and data normalized to the internal reference material.

Third Stage

a. Collection and analysis of all data.

The internal reference material chosen was H. C. Starck (LC-10) silicon nitride (Si_3N_4). Besides being used for normalization, it was also used for instrument calibration. Particle size distribution of this reference material was reported with all of the powders, even if this normalization technique was not used.

Empirical Normalization

Empirical equations (Hatch-Choate) were available to calculate any diameter (e.g., weight diameter) from any other diameter (e.g., volume diameter) using the measured diameter and standard deviation of the size distribution. This could be built into the data analysis stages if this method is the chosen technique for comparability.

Recommended Characterization Parameters and Reporting Formats

The following characterization methods were recommended for obtaining the desired parameters for powder characterization.

Physical

1. Particle Size Distribution: same and multiple techniques*
2. Surface Area: single/multiple point*
3. Permeametry Number: Fisher Sub Sieve Sizer (FSSS)
4. Bulk Properties:
   (a) Density: pycnometer density/tap density
   (b) Flowability: shear
5. Thermal: DTA/TGA
6. Morphology: SEM

Chemical (Bulk Concentrations)

1. Major Element Content
2. Minor Element Content
3. Trace Element Content

*As many techniques as participants want to utilize, but reporting on each one.

4. Surface Chemistry: ESCA/Auger/FTIR
5. Phase Composition

Defect Evaluation

1. Agglomeration Factor (definition to be determined)
2. Morphology: SEM (aspect ratio, circularity)
3. Residual Stress

A **full** characterization procedure log was kept, including the details of sample preparation, standard operating procedures, and standards (other than program reference) used.

DATA MANAGEMENT

Reporting and Assembly of All Data

The preliminary data reporting formats were agreed upon by all participants and appear on Tables 3 through 7. As soon as possible, the participants will exchange the test data in the adopted format for both the internal reference material (Starck Si_3N_4) and the first primary powder. The results will be reported and discussed at technical workshops, as agreed upon by the participants.

Table 3. PHYSICAL CHARACTERISTICS - SIZE & DISTRIBUTION

Powder:

Lab:

I.D. #	Modes	$D_g(1)$	$\sigma_g(1)$	$\%[d_g(1)]$	$d_g(n)$	$\sigma_g(n)$	$\%[d_g(n)]$	Method*	Normt	Std. Values

*Sample preparation/method summary, powder density assumed or measured
†Values normalized to selected reference material

Table 4. PHYSICAL CHARACTERISTICS - MORPHOLOGY

Powder:

Lab:

I.D. #	Sw		Theor. Density X-Ray Density	Permeametry #		Sw(meas.) Sw(calc.)	Aspect Ratio Long/Short	Other
	Value	Std. Used		Value	Std. Used			

Sample preparation/method summary

Table 5. CHEMICAL CHARACTERISTICS - SUMMARY

Powder:

Lab:

	% Major				% Minor				% Trace Volatiles				% Trace Nonvolat.				
I.D. #	A	B	§*	Total	a	b	§	Total	m	n	§	Total	v	w	§	Total	Stds

*As many constituents as needed
Sample preparation/method summary

Table 6. CHEMICAL CHARACTERISTICS - PHASES

Powder:

Lab:

		% Crystalline			
I.D. #	% Noncrystalline	A	B	C	Foreign Matter*

*Loss on ignition to 115°C
Sample preparation/method summary

Table 7. PHYSICAL DEFECTS

Powder:

Lab:

I.D. #	Agglomeration Factor	Residual Stress	Other - 1	Other - 2

Sample preparation/method summary

Statistical Analysis of Round Robin Data

The powder characterization undertaken by participants in IEA/Annex II included physical and chemical measurements, determination of phase composition, and identification of material defects. This interlaboratory comparison currently in progress will provide a comparison of current technology used for the evaluation of ceramic powders and will seek to identify areas in which better analytical standards and standard procedures are required. A variety of graphical and statistical methods will be used to display and evaluate the following five attributes: material, laboratory, characteristic, measurement method (including procedure), and sampling. The analysis will seek to identify the set of parameters which provide a nonredundant and unique description of the material for the characterization method which best measures each of the parameters. Significant differences in data with respect to laboratory and procedures will be indicative of areas in which better analytical standards and standard procedures are required.

PARTICIPANTS AND AGREED ASSIGNMENTS

The participating contracting parties were as follows:

1. The Kernforschungsanlage Juelich GmbH (KFA): Federal Republic of Germany

2. Styrelsen Teknisk Utveckling (STU): Sweden

3. The United States Department of Energy (DOE): United States of America

At the onset of Subtask 2 (Powder Characterization Studies), Dr. James W. McCauley of the U.S. Army Materials Technology Laboratory (T. Resetar, alternate) served as chairman, initiating and organizing the program. Currently, the leadership is in a transition phase. In future phases, as the program proceeds from technical survey and preliminary powder measurements and analysis to more detailed analysis and standard procedures, and formalization of data handling and analysis, Dr. Steve Hsu of the National Institute of Standards and Technology (A. Dragoo, alternate) will serve as chairman with J. W. McCauley and T. Resetar as advisors.

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